STUDY ON SILICON CARBIDE PRODUCED FROM RICE HUSK AS A REINFORCING AGENT

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology

In

Metallurgical & Materials Engineering

By

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BHAGIRATHI SINGH (107MM032)

Department of Metallurgical & Materials Engineering

National Institute of Technology

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NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

CERTIFICATE

This is to certify that the thesis entitled, " **STUDY ON SILICON CARBIDE PRODUCED FROM RICE HUSK AS A REINFORCING AGENT** " submitted by **Sidheshwar Kumar (107MM024)** and **Bhagirathi Singh (107MM032)**, in partial fulfillments for the requirements for the award of **Bachelor of Technology Degree** in **Metallurgical and Materials Engineering** at National Institute of Technology, Rourkela is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

Date:

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ABSTRACT

Now days, agricultural waste material are getting a wide exposure to the future generation of material science world. Rice husk is one of them, and its global production is approximately 140 million tons, annually. In the majority of cases much of the husk produced from the processing of rice is either burnt or dumped as a waste.

In the present work, an attempt has been made to synthesize the silicon carbide from rice husk and consolidate the prepared silicon carbide with aluminum matrix and form a composite by powder metallurgy route, this treatment of rice husk will act as a 'resource' for energy production and simultaneously avoids its disposal problems. Following the powder synthesis and consolidation with aluminum powder and sintering done at temperature of 550^0 C with holding time of 1 hr., extensive efforts have been undertaken for characterization by X-ray diffraction, SEM and EDX, microstructural evolution by optical microscopy and other analysis as well as assessment of mechanical properties like hardness under different loading conditions and wear behavior variables like time, wear depth and sliding distance. The results suggest that preparation of metal matrix composites of Al-SiC followed by conventional sintering method is a potential way for synthesizing new high strength commercial MMCs by utilization of biomass resources.

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CHAPTER 1

INTRODUCTION

1. INTRODUCTION

1.1 RICE HUSK

Rice husks (or rice hulls) are the hard protecting coverings of grains of rice. Rice husk is an agricultural residue abundantly available in rice producing countries. Husk Produced is around 20% of total rice production. The annual rice production in India and the World is 150 MTs (Million Tons) and 700 MTs, respectively, and hence husk produced in India and the World, the amounts are generally approximately in the range of 30 MTs and 140 MTs, respectively. Rice husk is generally not recommended as cattle feed since its cellulose and other sugar contents are low means there is limited source of energy for body. Furfural and rice bran oil are extracted from rice husk. Industries use rice husk as fuel in boilers and for power generation. Among the different types of biomass used for gasification, rice husk has a high ash content varying from 18 – 20 %. Silica is the major constituent of rice husk ash and the following tables gives typical composition of rice husk and rice husk ash. With such a large ash content and silica content in the ash it becomes economical to extract silica from the ash, which has wide market and also takes care of ash disposal. The dry analysis of rice husks are as follows:

Element Analysis	Mass Fraction %
Carbon	41.44
Hydrogen	4.94
Oxygen	37.32
Nitrogen	0.57
Silicon	14.66
Potassium	0.59
Sodium	0.035
Sulfur	0.3
Phosphorous	0.07
Calcium	0.06
Iron	0.006
Magnesium	0.003

Table 1.1 Chemical analysis of rice husks

Compositions	Mass Fraction (%)
Silica (SiO2)	80 – 90
Alumina (Al2O3)	1 – 2.5
Ferric oxide (Fe2O3)	0.5
Calcium oxide (CaO)	1 – 2
Magnesium oxide (MgO)	0.5 – 2.0
Sodium oxide (Na2O)	0.2 – 0.5
Potash	0.2
Titanium dioxide (TiO2)	Nil
Loss on Ignition	10 – 20

Table 1.2 Compositional analysis of rice husks

1.2 SILICON CARBIDE

Silicon carbide (SiC), also known as carborundum, is a compound of silicon and carbon with chemical formula SiC. It occurs in nature as the extremely rare mineral moissanite. Grains of silicon carbide can be bonded together by sintering to form very hard ceramics which are widely used in applications requiring high endurance, such as car brakes, car clutches and ceramic plates in bulletproof vests. Silicon carbide with high surface area can be produced from SiO_2 contained in plant material [12]. Silicon Carbide is potentially useful such as high temperature structure material, because of its high hardness, high oxidation resistance, good thermal shocking resistance etc. However, the production of high density ceramics by solid state sintering is difficult due to the strong covalent nature of Si-C bond. The solid state sintering of SiC can be performed at high temperature up to 2200^0 C.

1.2.1 NATURAL OCCURRENCES

Moissanite is natural form of Silicon carbide. When the first artificial moissanite reached the jewelry market, it has been regarded as an excellent diamond substitute, with optical properties exceeding those of diamond. Because it has its own unique appearance, it cannot be truly called a diamond stimulant [8] [10]. Its ethical production, however, does make it a popular alternative to diamonds.

Because of its hardness, it is useful for high-pressure experiments (e.g., using diamond anvil cell) competing there with diamond. Large diamonds, used for anvils, are prohibitively expensive. Therefore for large-volume experiments, much cheaper synthetic moissanite is a more realistic choice. Synthetic moissanite is also interesting for electronic and thermal applications because its thermal conductivity is similar to that of diamonds. High power SiC electronic devices are expected to play an enabling and vital role in the design of protection circuits used for motors, actuators, and energy storage or pulse power systems [13] [9] [18].

1.2.2 PRODUCTION

Due to the rarity of natural moissanite, silicon carbide is typically man-made. Most often it is used as an abrasive, and more recently as a semiconductor and diamond simulant of gem quality. The simplest manufacturing process is to combine silica sand and carbon in an Acheson graphite electric resistance furnace at a high temperature, between 1600 and 2500 °C [15] [6]. Fine SiO_2 particles in plant material (e.g. rice husks) can be converted to SiC by heating in the excess carbon from the organic material.

1.2.3 STRUCTURE AND PROPERTIES

Silicon carbide exists in about 250 crystalline forms. The polymorphism of SiC is characterized by a large family of similar crystalline structures called polytypes. They are variations of the same chemical compound that are identical in two dimensions and differ in the third. Thus, they can be viewed as layers stacked in a certain sequence. Alpha silicon carbide (α-SiC) is the most commonly encountered polymorph; it is formed at temperatures greater than 1700 °C and has a hexagonal crystal structure (similar to Wurtzite). The beta modification (β-SiC), with a zinc blende crystal structure (similar to diamond), is formed at temperatures below 1700 °C [8].

Properties	**Structure-3C (β)**	**Structure-4H**	**Structure-6H (α)**
Crystal structure	Zinc blende (cubic)	Hexagonal	Hexagonal
Lattice constants (Å)	4.3596	3.0730; 10.053	3.0730; 15.11
Density (g/cm^3)	3.21	3.21	3.21
Bandgap (eV)	2.36	3.23	3.05

Bulk modulus (GPa)	250	220	220
Thermal conductivity (W/(cm·K))	3.6	3.7	4.9

Table 1.3 Properties of major silicon carbide polytypes

Pure SiC is colorless. The brown to black color of industrial product results from iron impurities. The high sublimation temperature of SiC (approximately 2700 °C) makes it useful for bearings and furnace parts. Silicon carbide does not melt at any known pressure [20]. It is also highly inert chemically. There is currently much interest in its use as a semiconductor material in electronics, where its high thermal conductivity, high electric field breakdown strength and high maximum current density make it more promising than silicon for high-powered devices [3] [12]. SiC also has a very low coefficient of thermal expansion (4.0×10^{-6}/K) and experiences no phase transitions that would cause discontinuities in thermal expansion.

1.2.4 USES OF SILICON CARBIDE

- Abrasive and cutting tools
- Structural material
- Automobile parts
- Jewelry
- Electric and electronic systems
- Thin film pyrometry and heating elements
- Carborundum printmaking
- Graphene production

1.3 METAL MATRIX COMPOSITE

The matrix in a metal matrix composite (MMC) is usually an alloy, rather than a pure metal, and there are three types of such composites [1]:

- Dispersion-strengthened, in which the matrix contains a uniform dispersion of very fine particles with diameters in the range 10–100 nm,
- Particle-reinforced, in which particles of sizes greater than 1 μm are present, and

- Fiber-reinforced, where the fibers may be continuous throughout the length of the component, or less than a micrometer in length, and present at any volume fraction.

Compared to monolithic metals, MMCs have:

- Higher strength-to-density ratios,
- Higher stiffness-to-density ratios,
- Better fatigue resistance,
- Better elevated temperature properties,
- Higher strength,
- Lower creep rate,
- Lower coefficients of thermal expansion, and
- Better wear resistance.

The advantages of MMCs over polymer matrix composites are [1] [2]:

- Higher temperature capability,
- Fire resistance,
- Higher transverse stiffness and strength,
- No moisture absorption,
- Higher electrical and thermal conductivities,
- Better radiation resistance,
- No out gassing,
- Fabric ability of whisker and particulate-reinforced MMCs with conventional metalworking equipment.

Some of the disadvantages of MMCs compared to monolithic metals and polymer matrix composites are [1] [3]:

- Higher cost of some material systems,
- Relatively immature technology,
- Complex fabrication methods for fiber-reinforced systems (except for casting),
- Limited service experience.

1.3.1 PROCESSING OF MMCs

Accordingly to the temperature of the metallic matrix during processing the fabrication of MMCs can be classified into four categories:

(a) Liquid phase processes,

(b) In – Situ Processes,

(c) Two phase (solid-liquid) processes.

(d) Solid state processes.

1.4 SINTERING

Sintering is a powder metallurgy route for the process of forming objects from a metal powder by heating the powder at a temperature below its melting point, until its particles adhere to each other. Sintering is a solid state process and traditionally used for manufacturing ceramic objects. Most of the material exhibit sintering at homologous sintering temperatures between 0.5 Tm – 0.8 Tm range. With higher temperature, longer time, or smaller particles, the bond grows more rapidly and densification becomes prominent.

1.4.1 STEPS IN SINTERING

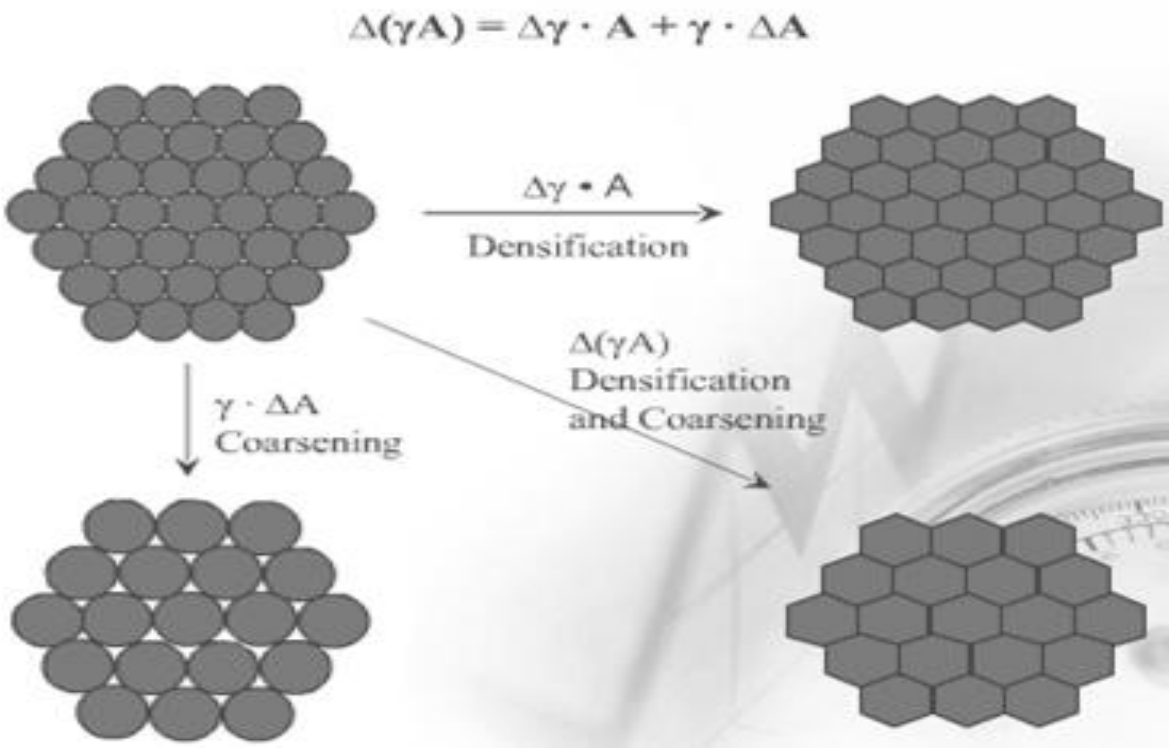

Figure 1.1 Ideal sintering steps

Where, $\Delta\gamma$ is change in surface energy and A is surface area.

The steps for sintering are as follows:

- Adhesion: This stage occurs when particle comes into contact.
- Rapid growth of the inter-particle neck.
- The pore structure becomes smooth and develops an interconnection, i.e. giving average larger grains with few grains.
- Pores are get closed due to grain growth.

Ideally, when two particle of diameter of D are compacted and then sintered then the resultant diameter of larger grain is 1.26*D [16]. These mentioned steps are shown in the below figure.

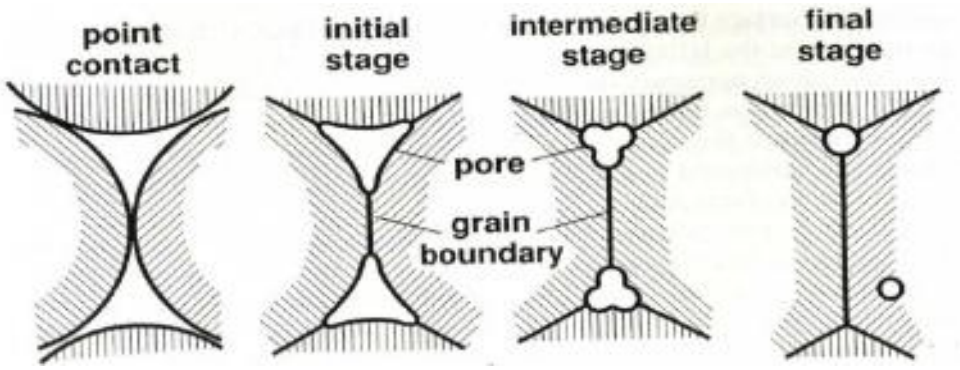

Figure 1.2 Sintering stages

The shown figure indicates that the pore structure changes during sintering, starting with particles in the point contact. The pore volume decreases and the pore become smoother. As pore spheroidization occurs, the pores are replaced by grain boundaries.

1.4.2 DENSIFICATION PARAMETER

Densification parameter is an important parameter for sintering operations. Ideally it should be positive but in some cases of sintering it is found negative. The positive value of densification parameter means there is increase in density due to sintering, i.e. sintered density should be higher than green density. It is followed by formula given below:

$$\textbf{Densification Parameter} = (\rho_s - \rho_g) / (\rho_{Th} - \rho_g)$$

Where, ρ_s is the sintered density of material, ρ_g is the green density of material, and ρ_{Th} is the theorectical density of material which has been calculated on ideal basis of material.

1.5 WEAR BEHAVIOUR

Perhaps the biggest in solving wear problems is that of anticipating the type(s) of wear to which components will be subjected. Material can be removed from a solid surface in only three ways: by melting, by chemical dissolution, or by the physical separation of atoms from the surface. The last method can be accomplished either by the one-time application of a high strain or by cyclic straining at lower magnitudes. Mechanical and chemical processes may operate separately or together, such as abrasion in a corrosive medium [1] [4] [14].

1.5.1 TYPES OF WEAR

Depending on the nature of movement of the media following types wear has been classified as:

- Abrasive wear,
- Sliding and Adhesive wear,
- Corrosive wear,
- Oxidational wear

1.5.2 EFFECT OF ENVIRONMENT ON ABRASIVE WEAR

In addition to the properties of a material, the environment affects wear. As stated earlier, abrasion loss rates are not intrinsic to a material. Environmental factors that affect abrasive losses include, but are not limited to: the type of abrasive and its characteristics, temperature, speed of contact, unit load of the abrasive on the material, humidity, and corrosive effects.

1.5.2.1 ABRASIVE

The hardness of the abrasive particles is important to the rate of abrasion of the subject material. As the hardness of the abrasive exceeds that of the wear material, abrasive wear typically becomes much worse .As the abrasive hardness exceeds the hardness of the material, it is able to penetrate the surface and cut/remove material without having its cutting edges broken or rounded.

1.5.2.2 TEMPERATURE

It might be expected that abrasive wear would increase as the temperature rises, because the hardness and yield strength decrease. Instead, for aluminum and copper, when the temperature was increased from ambient to 673 K, very little change in the abrasive wear rate was observed. It has been proposed that the reason for this small change is that during abrasion, small areas are adiabatically heated. At higher initial temperatures, the metal flow stress is reduced. This results in less heating in the material during the abrasion process. The end result is that areas around the

material that is being removed have a similar temperature, independent of starting temperature, and similar abrasion rates [2] [19].

1.5.2.3 SPEED OF CONTACT

The rate of abrasive wear has been found to slightly increase with increasing speed in the range from 0 to 2.5 m/s (0 to 8.2 ft/s). This increase in wear may be attributable to frictional heating. The effect is small, because all of the abrasion occurs in a near-adiabatic process. This should result in nearly the same peak temperature rise, independent of speed, for the tiny volume of material where the asperities are removing the material [4].

1.5.2.4 LOAD

When load causes fracture of abrasive particle then wear can increase. If the abrasive particle points are rounded, wear will decrease.

1.5.2.5 HUMIDITY

The effect of atmospheric humidity on abrasive wear is far from clear, and contrary results exist. Larsen- Basse studied the effect of atmospheric humidity on abrasive wear for a variety of pure metals and steels. When using SiC abrasive, wear usually increased with increasing humidity, up to 65% relative humidity. This increase is attributed to a moisture-assisted fracture of the SiC abrasive particle, which resulted in fresh sharp edges to cut into the surface of the material.

CHAPTER 2

LITERATURE SURVEY

2. LITERATURE SURVEY

[1] **C. Srinivasa Rao and G. S. Upadhyaya** [6] have studied on the processing and mechanical properties of 2014 and 6061 Al alloy-based power metallurgy composites containing up to 8 ~01% of SIC in either particle or continuous fibre form. For consolidation of the green compacts, liquid phase sintering under vacuum at 635°C was adopted. The addition of reinforcement imparted improved densification such that particulate composites were better densified than the fibrous ones. Relatively higher work hardening rates were observed in fibrous composites than in the particulate ones. The ductility values of obtained porous particulate composites were similar to those of the fully dense once reported in the literature.

[2] **Huei-Long Lee, Wun-Hwa Lu and Sammy Lap-Ip Chan** [7] have studied on the effects of sintered porosity, volume fraction and particle size of silicon carbide particles (SiC& on the abrasive wear resistance of powder metallurgy (P/M) aluminium alloy 6061 matrix composites. Aluminium alloy 6061 manufactured following the same route was also included for direct comparison of alloy 6061 manufactured following the same route was also included for direct comparison. The results show that the beneficial effect of hard Sic, addition on wear resistance far surpassed that of the sintered porosity in the P/M composites. The wear rates of composites decreased as the amount of Sic, increased. Aluminium alloy composites reinforced with larger SiC, size are more effective against abrasive wear than those reinforced with smaller SiC, size. The wear rates of the P/M aluminium alloy and 10 vol.% SiC, composite were significantly affected by the aging conditions. However, for the 20 vol.% SiC, composite, and for the composites with large SiC, size, the aging effect on the wear rate of the composites was found to be small.

[3] **Lauri Kollo, Marc Leparoux, Christopher R. Bradbury, Christian Jäggi, Efraín Carre˜no-Morelli, and Mikel Rodríguez-Arbaizar** [8] have studied that planetary milling for nano-silicon carbide reinforced aluminium metal matrix composites. High-energy planetary milling was used for mixing aluminium powders with 1 vol.% of silicon carbide (SiC) nano-particles. It was shown that mixing characteristics and reaction kinetics with stearic acid as process control agent can be estimated by normalised input energy from the milling bodies. For this, the additional parameter characterizing the vial filling was determined experimentally. Depending on the ball size, a local minimum in filling parameter was found, laying at 25 or 42% filling of the vial volume for the balls with diameter of 10 and 20mm, respectively. These

regions should be avoided to achieve the highest milling efficiency. After a hot compaction, fourfold difference of hardness for different milling conditions was detected. Therewith the hardness of the Al–1 vol.% nano SiC composite could be increased from 47HV0.5 of pure aluminium to 163HV0.5 when milling at the highest input energy levels.

[4] **P. K. Mishra, B. C. Mohanty, and B. B. Nayak** [9] have studied that thermal plasma synthesis of silicon carbide from boiler burnt rice husk. Thermal plasma technology has been utilized for synthesis of powder silicon carbide, the burnt rice husk and wood charcoal being processed in a different way. The product has been characterized by chemical analysis, XRD, SEM and also particle size analyser. The yield was 60% which produced both α and β silicon carbide. Based on microstructural study a mechanism has been proposed for formation through liquid phase of SiC.

[5] **U. Kalapathy, A. Proctor, J. Shultz** [10] have studied that rice hull ash (RHA), a waste product of the rice industry is its husk. The method based on alkaline extraction followed by acid precipitation was developed to produce pure silica xerogels from RHA, with minimal mineral contaminants. The silica gels produced were heated to 80^{o}C for 12 hr to obtain xerogels. Silica and mineral contents of xerogels were determined by energy dispersive X-ray (EDX) and inductively-coupled plasma (ICP) emission spectrometers, respectively. Xerogels produced from RHA had 93% silica and 2.6% moisture. The major impurities of silica produced from RHA at an extraction yield of 91% were Na, K, and Ca. Acid washing prior to extraction resulted in silica with a lower concentration of Ca (<200 ppm). However, final water washing of the xerogel was more reactive in producing silica with lower overall mineral content (Na< 200 ppm and K< 400 ppm). X-ray diffraction patterns revealed the amorphous nature of silica xerogel.

[6] **Kannigar Dateraksa, Kuljira Sujirote, and Pimpa Limthongkul** [11] have studied on pressureless sintering of nano-silicon carbide from rice husk. Silicon carbide obtained from direct pryolysis of raw rice husk exhibited very fine microstructure, its sintering behavior may resemble that of nano-silicon carbide. After milling the isostatic compaction done at 250 MPa and then sintered at 1850^{o} C for 6 hours and all analysis have been carried out. It is found that prolonged sintering even at room temperature led at extensive grain growth. On the other hand, SiC from rice husk which although composed of small grain SiC, but those grains are connected together in the form of large agglomerate, therefore, required higher sintering temperature and native porous structure of the rice huskstill remained for some extent after sintering.

CHAPTER 3

EXPERIMENTAL DETAILS

3. EXPERIMENTAL DETAILS

3.1 FORMATION OF SILICON CARBIDE FROM RICE HUSK

Rice husks (RH) were collected from a local rice mill with the husk dimension of 7-10 mm long, 1.5-2.0 mm wide and 0.10-0.15 mm thick. Dry raw rice husks were washed thoroughly with water to remove the adhering soil and other contaminations present in them and then dried in the sunlight for 24 hrs. These were designated as raw rice husk (RRH).

The chemical analysis of these washed RRH was carried out as shown in the flow chart. Acid leaching was performed by treating the RRH with dilute HCl (1:1) in distilled water for 1 hour. It was then washed thoroughly with distilled water followed by treatment with dilute ammonia solution (10 vol. %) for 1 hr to remove the traces of acid. The treated husk was taken out and washed thoroughly in distilled water followed by drying in air for 24 hrs. About 5 g samples of RRH and acid-treated rice husk (ARH) were taken in separate cylindrical alumina crucibles and introduced into a muffle furnace for pyrolysis at different temperatures varying from 700^0 C to 1100^0 C at an interval of 100^0 C for 2 hrs. Soaking time in static air, and weight losses were measured after each pyrolysis by an electronic balance. Black ash (BA) and White ash (WH), both the powders were prepared for further study.

3.2 XRD ANALYSIS

The XRD analysis conducted to detect the phases present in the silicon carbide and white ash, which have been made from rice husk were studied by X-ray diffraction using a high resolution PHILIPS X'Pert High Score program system, with Cu - Kα radiation (λ=1.54 A). The phases formed were identified by insidious comparison of the recorded diffraction peaks with the ICDD database. Instrumental broadening was correlated by using FWHM of Silicon standard. X-ray Diffraction scans were conducted for phase present in the produced material.

Parameters	Value
Raw Data Origin	PHILIPS-binary (scan)
Start Position [°2Th.]	10.0100
End Position [°2Th.]	79.9900
Step Size [°2Th.]	0.0200
Scan Step Time [s]	0.6000
Scan Speed	2° (degree) per minute
Scan Type	Continuous

Anode Material	Cu
Voltage applied	30 kV
Current Flow	20 mA

Table 3.1 List of parameter used during XRD analysis

3.3 SCANNING ELECTRON MICROSCOPY

Microstructural characterization studies were conducted to examine distribution of reinforcement throughout the matrix. This is accomplished by using scanning electron microscope. Silicon carbide produced from rice husk, white ash and sintered composites of Al-1% SiC and pure aluminum samples were metallographically polished prior to examination. Characterization is done in etched conditions. Etching was accomplished using Keller's reagent. The SEM micrographs of composites were obtained using the scanning electron microscope. The images were taken in secondary electron (SE) mode. This analysis was done by a JEOL 6480 LV scanning electron microscope (SEM) equipped with an energy dispersive X-ray (EDX) detector of Oxford data reference system.

Figure 3.1 JEOL JSM-6480LV scanning electron microscope

Parameters	Value
Detector	Silicon
Accelerating voltage	15.00 (kV)
Process time	5 seconds
Magnification	1000 X, 2500 X, 5000 X, 10000 X, 20000 X

Table 3.2 Parameters used during SEM analysis

3.4 CONSOLIDATION AND SINTERING

The pure aluminum powder of size 150µm has been taken and silicon carbide, made from rice husk is mixed in mortar and then the mixed they were compacted by cold pressing hydraulic machine of 10 mm diameter stainless steel dies with load of 3.2 tons, i.e. equivalent pressure is 400 MPa. The powders used for making cold compact weighs around 1.1 grams. The cold compact is then sintered in a muffle furnace 10^0 C/min with slow heating rate up to at a temperature of 550^0 C with the holding time of 1 hour.

Parameters	Pure Aluminum	Al-1%SiC Composite
Temperature	25^0 C	25^0 C
Particle size of Al powder	150 µm	Not Calculated
Diameter of Die	10 mm	10 mm
Load Applied	400 MPa	400 MPa
Compaction Time	5 minutes	5 minutes
Weight of Green Pellet	1.0415 g	0.9050 g
Weight of Sintered Pellet	1.1003 g	0.9637 g

Table 3.3 Parameters used during compaction

Material	Theoretical Density (g/cm3)	Green Density (g/cm3)	Sintered Density (g/cm3)	Densification Parameter
Pure Aluminum	2.7000	2.6521	2.6641	0.2505
Al-1%SiC	2.7042	2.6524	2.6643	0.2297

Table 3.4 Comparison of different densities

3.5 HARDNESS MEASUREMENT

The specimens for hardness measurement were ground flat using a belt grinder. Sufficient care was taken so that the opposite surfaces were flat and parallel to each other to ensure the accuracy of measurements. Vickers microhardness tests were carried out on the Vickers Hardness testing machine. The machine was calibrated using a standard test block of known hardness prior to the hardness tests [3] [4]. At least ten readings were taken from each sample to get the mean value. The microhardness was measured at minimum loads of 300 gf, 500 gf, and 1000 gf with a dwell time of 10 seconds. The Vickers hardness has been calculated using expression:

$$H_V = \frac{1.854P}{d_{avg}{}^2}$$

Where, P is load in kgf (Kilogram Force) and $d_{avg} = \frac{d_1 + d_2}{2}$; where, d_1 and d_2 are the lengths of the and two indentation diagonals in millimeters. This hardness testing was done by a LECO-LV700, Vickers Hardness Indenter having loading capacity range from 300 gf to30 kgf.

Figure 3.2 Vickers hardness testing machine

3.6 WEAR TESTING

A DUCOM–TR-208-M1, Ball on Plate Wear Tester, Bangalore made computerized pin on- disc wear test machine was used for these tests. The wear testing was carried out at a different sliding velocity with different normal loads. A cylindrical pin of size 1.1cm diameter and 2.1cm length, prepared from composite casting, was loaded through a vertical specimen holder against horizontal rotating disc. The rotating disc was made of Hardened carbon steel ball of diameter 50 mm and hardness of 64 HRC. The weights were measured before and after each test segment to determine the abrasive wear loss of each sample.

Parameters	Value
Load Applied	500 g
Speed	10 rpm
Track Diameter	2 mm
Ball Diameter	4 mm
Testing Time	5 minutes

Table 3.5 Parameters used during Vickers hardness testing

CHAPTER 4

RESULT AND DISCUSSION

4. RESULT AND DISCUSSION

4.1 XRD – ANALYSIS

The phase analysis of silicon carbide (SiC) and white ash, which have been produced from rice husk. The figures shown below are the XRD plots of both SiC and white ash. In the plot SiC all the peaks are sharper, i.e. it represents the silicon carbide is in crystalline form and crystal sizes have been calculated by the help of modified Scherrer formula and crystal size found in the range of 118nm - 50nm. During phase analysis, it is found that all the sharper peaks are of SiC. But, in the plot of white ash, there are one sharper peak of silicon oxide, i.e. material is almost amorphous. The particle size of white ash is found in the range of 207nm – 35nm.

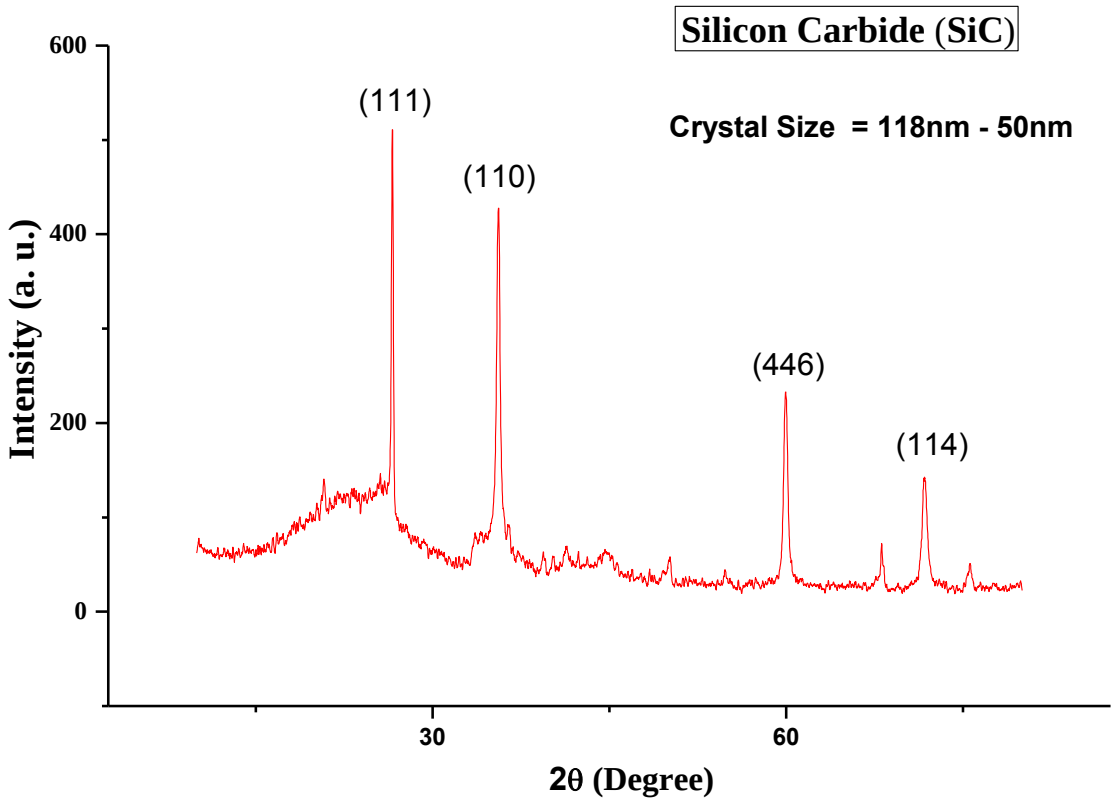

Figure 4.1 XRD plot of SiC produced from rice husk

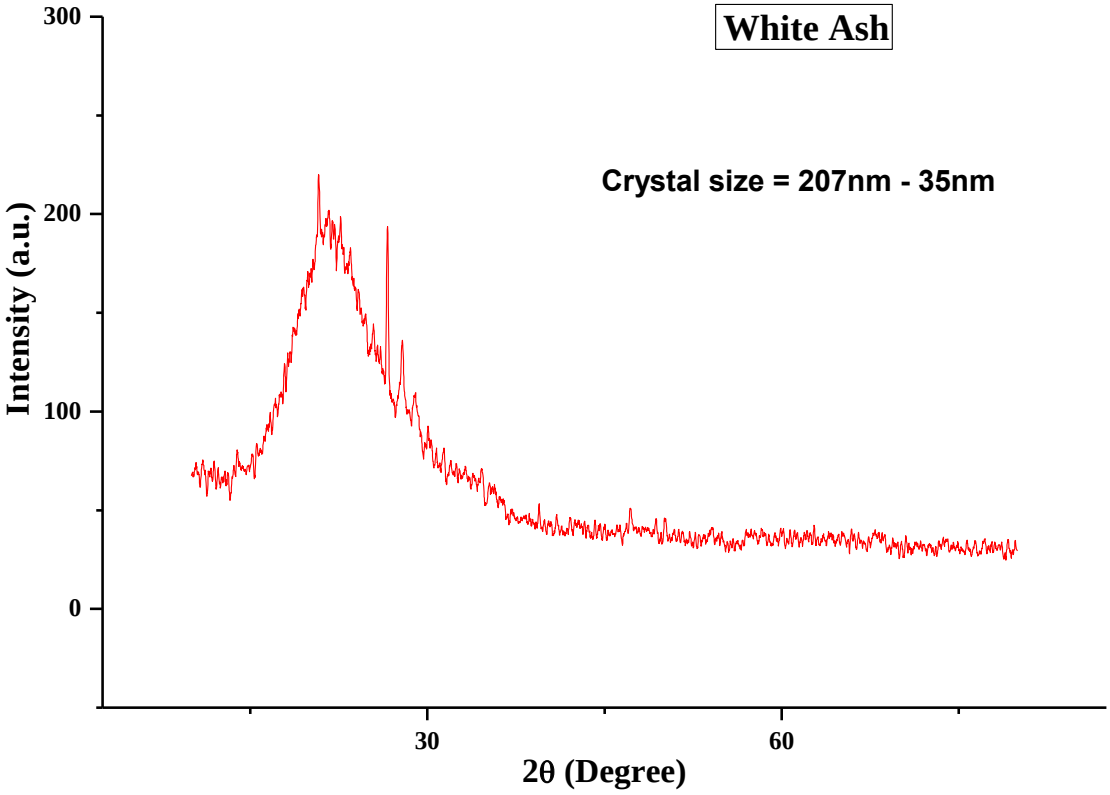

Figure 4.2 XRD plot of white ash produced from rice husk

4.2 SCANNING ELECTRON MICROSCOPY

The chemical analysis of silicon carbide (SiC) and white ash, which has been produced from rice husk, were studied under SEM (Scanning Electron Microscope) and further chemical analysis has been done with the help of EDS (Energy Dispersive Spectroscopy).

4.2.1 SILICON CARBIDE

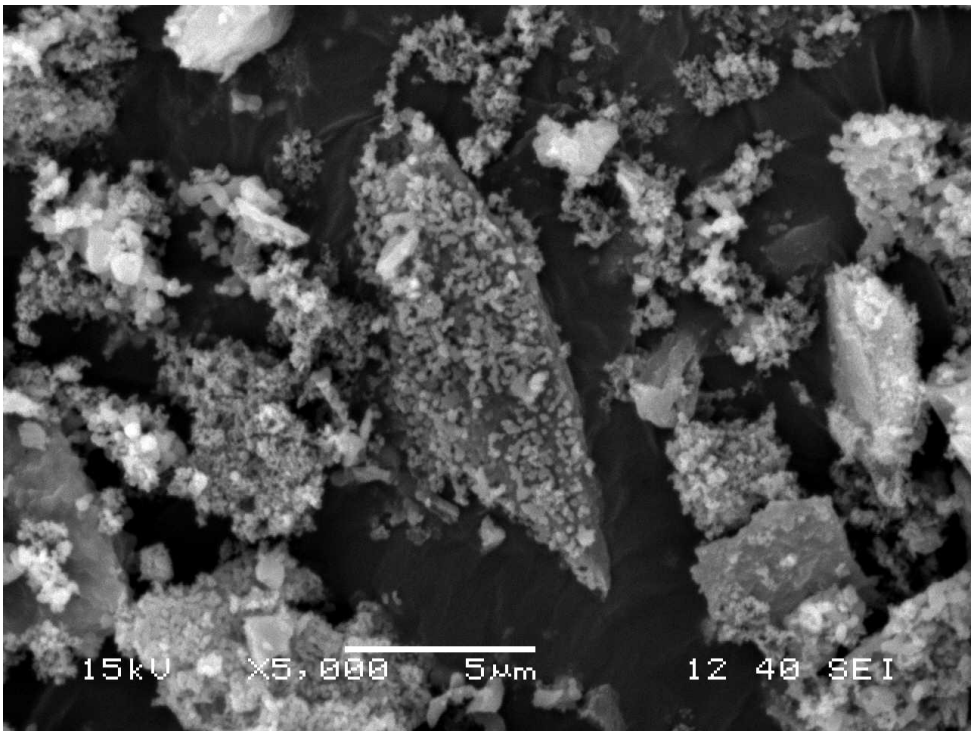

Figure 4.3 Scanned image of silicon carbide particles

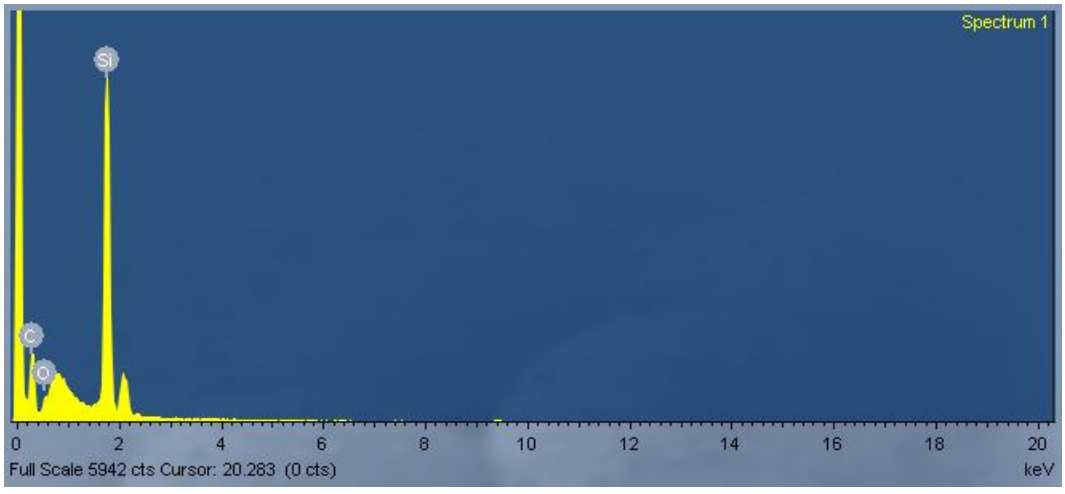

Figure 4.4 EDX pattern of silicon carbide particle

Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	11.49	0.3700	57.95	1.19	74.38
O K	1.76	0.5344	6.14	1.09	5.92
Si K	21.02	1.0922	35.90	0.96	19.71
Totals			100.00		

Table 4.1 Elemental analysis silicon carbide particle

4.2.2 WHITE ASH

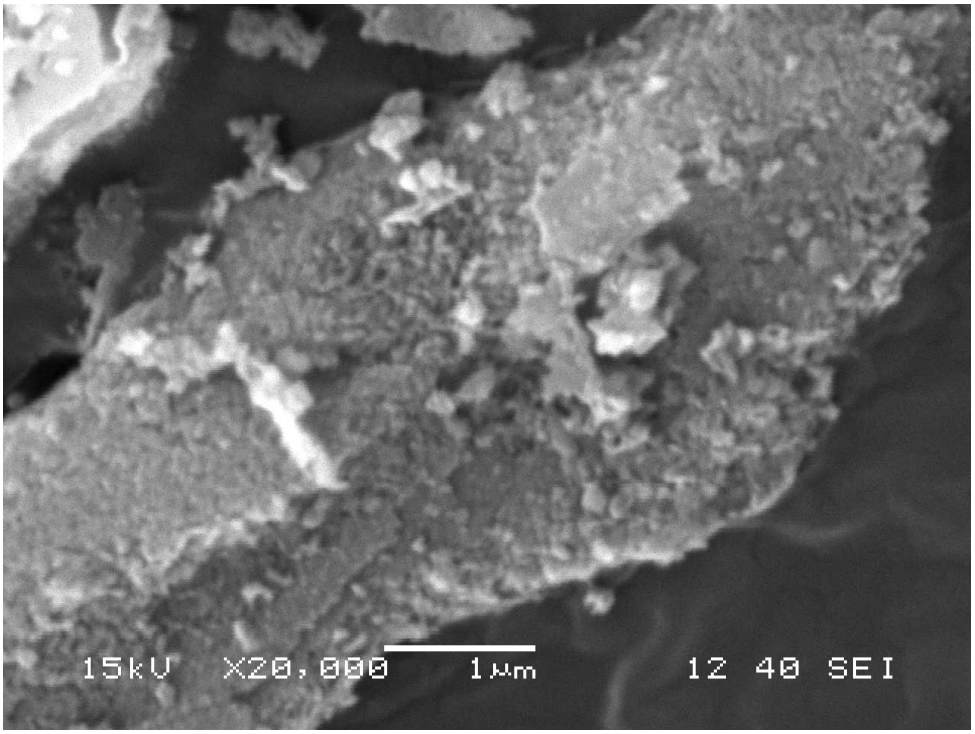

Figure 4.5 Scanned image of white ash particle

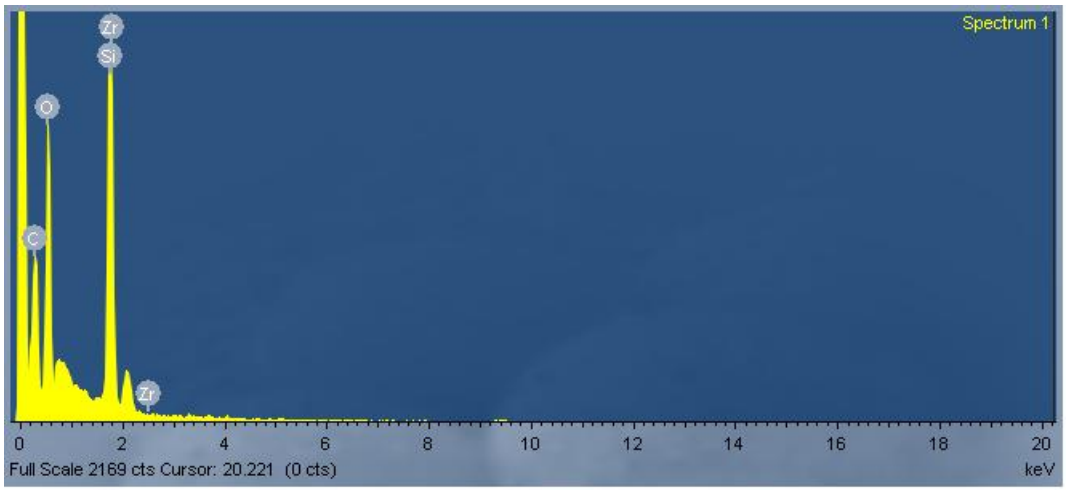

Figure 4.6 EDX pattern of white ash

Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	12.86	0.4956	30.91	1.49	41.94
O K	33.96	0.8764	46.12	1.19	46.98
Si K	14.78	1.0138	17.36	0.50	10.07
Zr L	3.25	0.6905	5.61	0.55	1.00
Totals			100.00		

Table 4.2 Elemental analysis of white ash

4.2.3 ALUMINUM-SILICON CARBIDE COMPOSITE

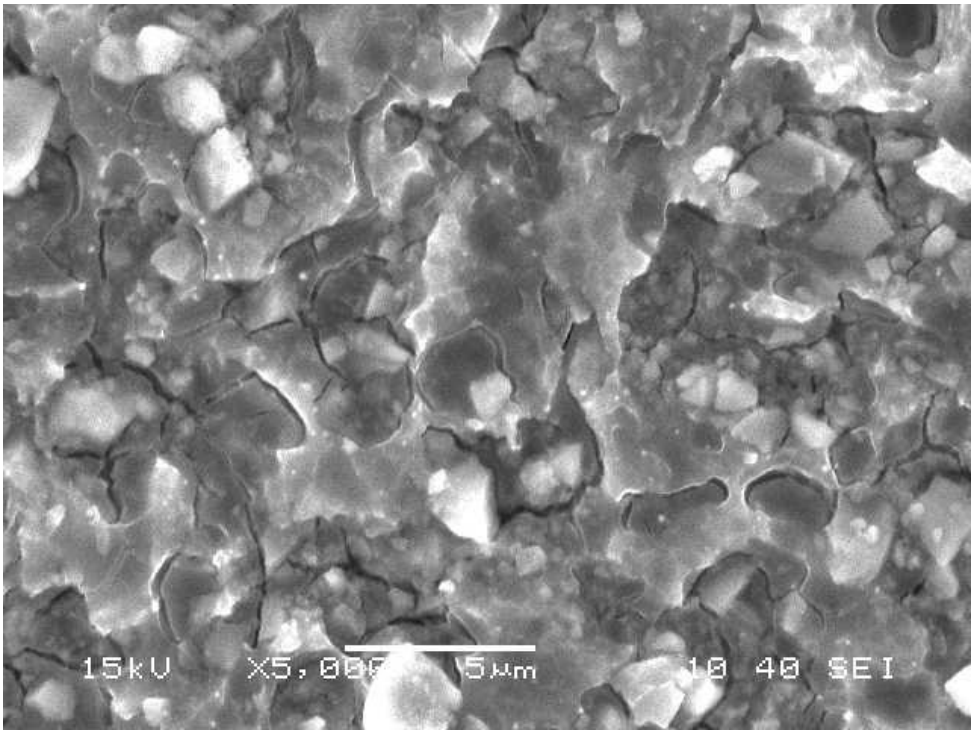

Figure 4.7 Scanned image of Al-SiC (1%) composite

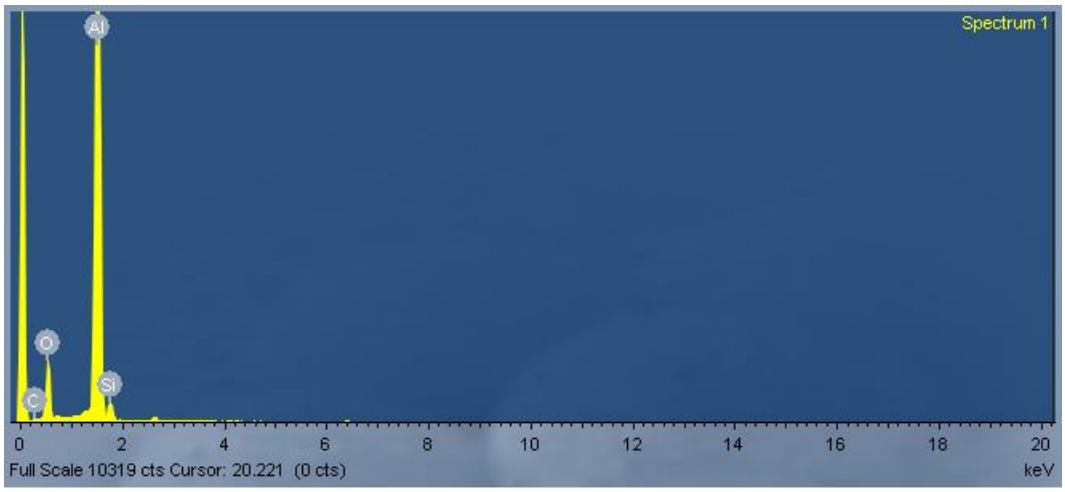

Figure 4.8 EDX pattern of Al-SiC (1%) composite

Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	0.59	0.2356	4.75	0.90	8.86
O K	11.51	1.0387	21.15	0.48	29.64

Al K	44.73	1.2008	71.01	0.79	59.02
Si K	1.06	0.6511	3.09	0.17	2.47
Totals			100.00		

Table 4.3 Elemental analysis of Al-SiC (1%) composite

4.2.4 PURE ALUMINUM

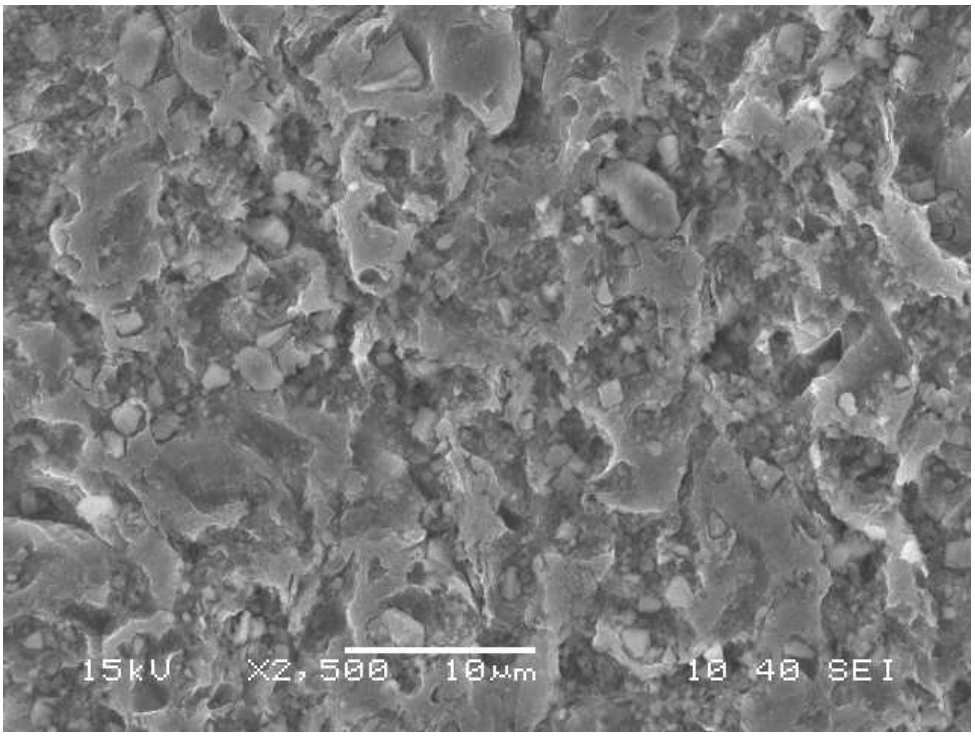

Figure 4.9 Scanned image of pure aluminum

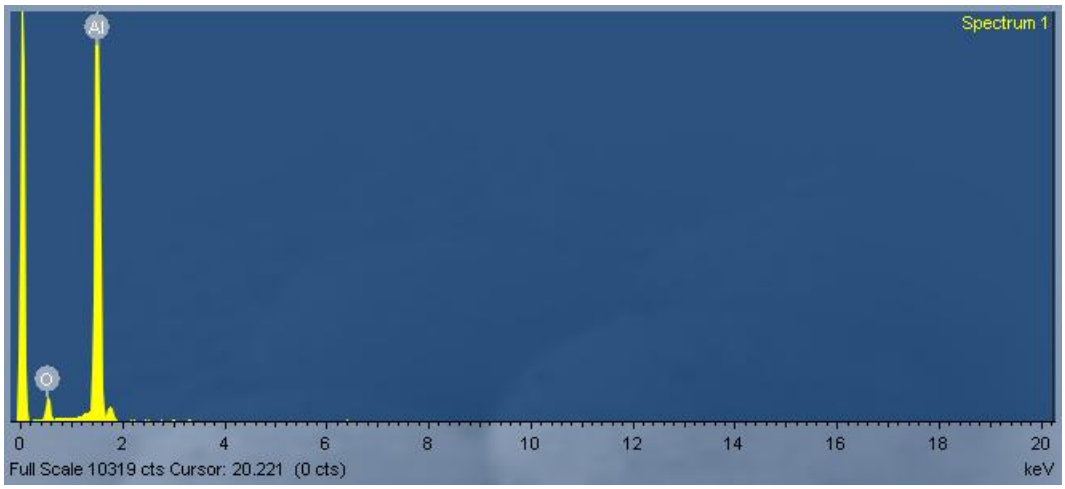

Figure 4.10 EDX pattern of pure aluminum

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
O K	7.38	1.0539	15.88	0.56	24.15
Al K	46.39	1.2513	84.12	0.56	75.85
Totals			100.00		

Table 4.4 Elemental analysis of pure aluminum

Above figures and tables of SEM, show that there the silicon carbide particles, which have been formed from rice husk and their chemical analysis by EDS also shows of silicon and carbon are present in the provided sample. The SEM analysis for sintered pellets of pure aluminum and Al-SiC (1%) composite have been done and shows the presence dispersed phase, i.e. silicon carbide is homogeneously distributed in the matrix phase of aluminum and on the surface of the pellets there are some white particles which are the traces of aluminum oxides (Al_2O_3), formed during sintering process with atmospheric oxygen and pores in the grain boundaries are clearly visible during the investigation.

4.3 HARDNESS MEASUREMENT

Applied Load (in g)	Diagonal (d_1, in µm)	Diagonal (d_2, in µm)	Vickers Hardness	Average Hardness
300 g	165.3	172.9	19.5	19.5
	165.6	172.7	19.4	
	164.9	172.1	19.6	
	165.2	173.4	19.4	
	164.5	173.1	19.5	
500 g	201.5	205.9	22.3	22.3
	201.9	206.1	22.2	
	202.4	205.1	22.3	
	202.0	205.8	22.3	
	201.7	206.5	22.3	
1000 g	269.4	271.5	25.4	25.3
	270.0	271.9	25.3	
	269.6	271.6	25.3	

	270.8	272.4	25.1	
	269.9	271.4	25.3	

Table 4.5 Hardness under different load for pure aluminum

Applied Load (in g)	Diagonal (d_1, in µm)	Diagonal (d_2, in µm)	Vickers Hardness	Average Hardness
300 g	151.0	149.8	24.6	24.6
	151.6	149.9	24.5	
	152.4	148.7	24.6	
	151.8	150.3	24.4	
	150.0	150.1	24.7	
500 g	176.8	176.8	29.7	29.7
	175.6	176.9	29.8	
	176.3	176.5	29.8	
	177.1	176.2	29.7	
	176.9	176.0	29.7	
1000 g	252.2	249.6	29.5	29.6
	250.8	248.2	29.8	
	251.1	249.1	29.6	
	253.2	247.9	29.5	
	250.0	248.7	29.8	

Table 4.6 Hardness under different load for Al-SiC (1%) composite

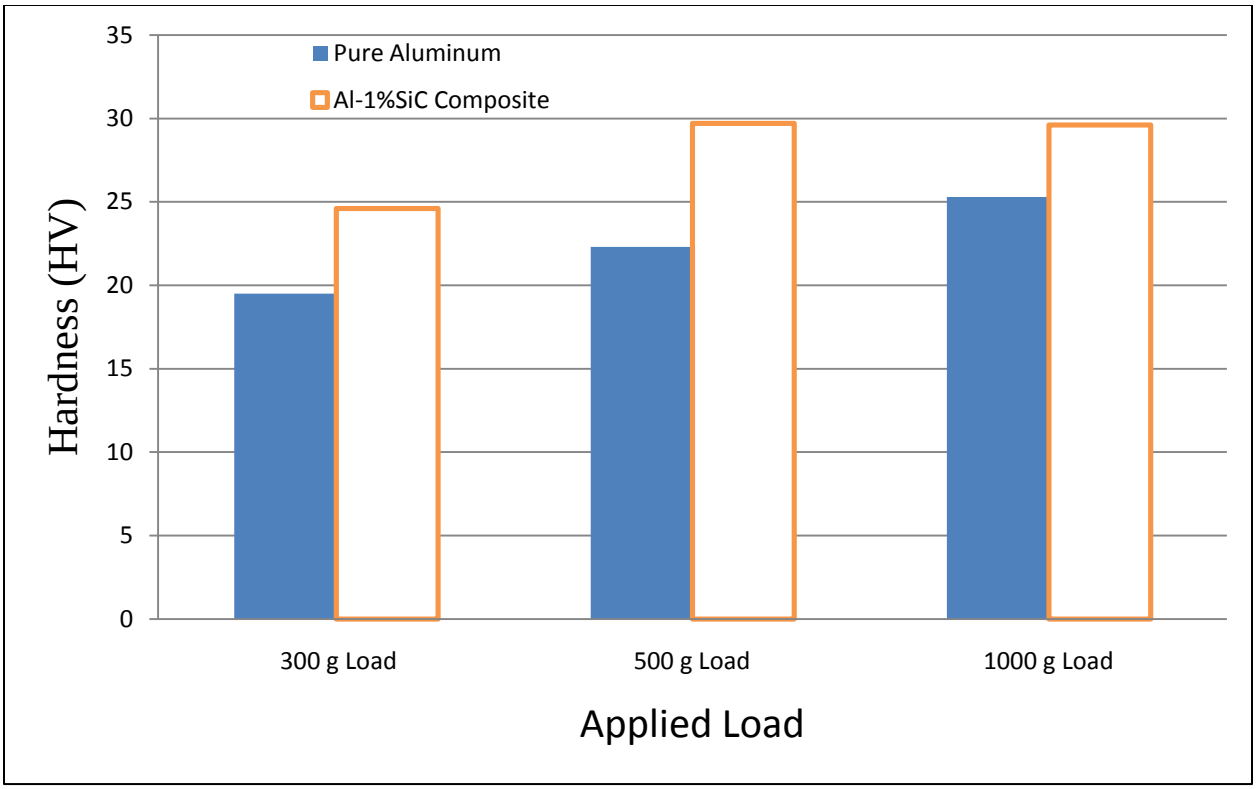

Figure 4.11 Comparison between pure Al and Al-SiC (1%) composite under different load

Above plot shows the relation between applied load and hardness value (in HV Scale) sintered pellets of pure aluminum and Al-SiC (1%) composite. It is seen that at low load, the hardness value of both pellets show comparatively lesser value than harness at high loading conditions. Another important observation is that hardness value of pure aluminum is lesser than hardness value of Al-SiC (1%) composite in different loading conditions means increase in hardness due to dispersed phase.

4.4 WEAR BEHAVIOR

4.4.1 EFFECT OF SLIDING DISTANCE ON WEAR DEPTH

Below figure shows the relation between sliding distance and wear depth of the composites at a constant load of 4.9 N. The above figure clearly shows that change of wear depth initially high and after a certain sliding distance the change of wear depth becomes constant. Like variation of wear depth with load in the present case also increase in dispersed phase silicon carbide reduces the wear rate.

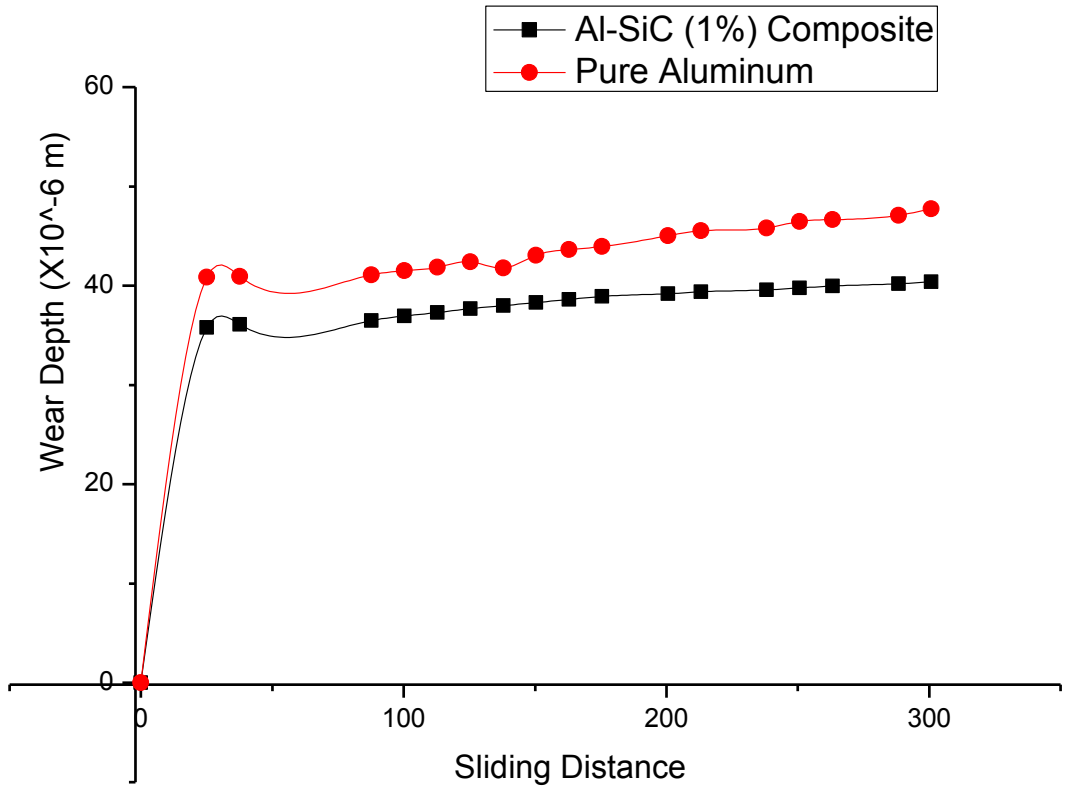

Figure 4.12 Plot between wear depth and sliding distance

4.4.2 EFFECT ON WEAR DEPTH WITH TIME

Below figure shows the relation between wear depth and time of the composites at a constant sliding velocity 10 rpm. It is seen that as time of wear increases, there is increase in wear depth and change in wear depth of composite is constant after sometime. The wear depth in case of as pure aluminum sample is higher than in Al-SiC (1%) composite.

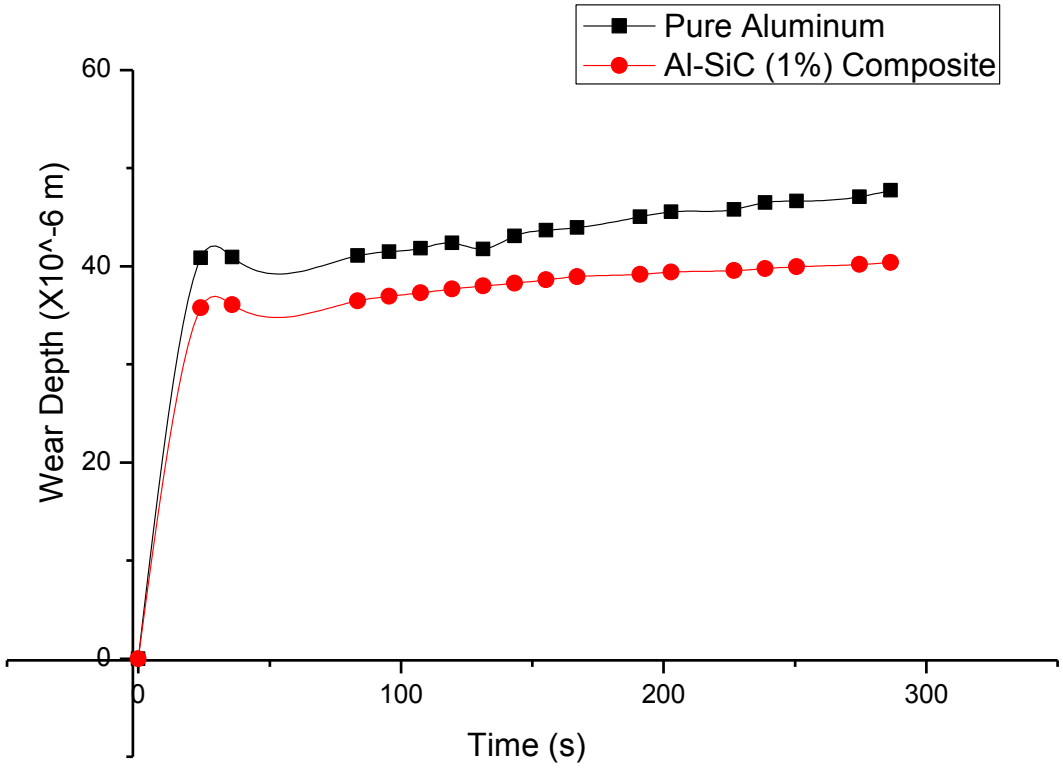

Figure 4.11 Plot between wear depth and time of sliding

CHAPTER 5

CONCLUSION

5. CONCLUSION

The following conclusion may be drawn from the present work:

- The silicon carbide (SiC) powder can be synthesized from rice husk.
- The prepared SiC powder is almost crystalline particle is appeared.
- The produced white ash powder is almost amorphous and some crystalline particle of silicon oxide appeared.
- The crystal size of prepared SiC powder is found to be 118-50 nm range.
- Cold compaction of pure Al and Al-SiC (1%) powders were done by hydraulic pressing machine at high pressure, 400 MPa.
- There is increase in density of cold compacted pellets after sintering and densification parameter is found to be 0.22-0.25.
- The hardness value of sintered pellet of pure aluminum and Al-SiC (1%) composite is found to be in the range of 19-25 HV and 25-30 HV.
- Strengthening of composites is due to particle reinforcement, dispersion strengthening and solid solution strengthening.
- The Al-SiC (1%) composite shows better wear resistance than pure Al, due to its superior load bearing capacity.
- The wear resistance has improved significantly by addition of SiC.

CHAPTER 6

FUTURE PLAN OF WORK

6. FUTURE PLAN OF WORK

The following points may be concluded for future plan of work:

- Silicon carbide produced from rice husk can be scaled up to 100g for the further analysis.
- As prepared silicon carbide powder can be kept in liquid nitrogen, alone then it can be mechanically ball milled for different timing and it's TEM, XRD, SEM and other characterizations can be done.
- As prepared silicon carbide powder can be mechanically alloyed with aluminum/stainless steel/copper or any other metal/alloys and it's TEM, XRD, SEM and other characterizations can be done to find out the nano-metric dispersion size distribution of SiC in the matrix.
- Above mentioned alloyed milled powders can be cold compacted at different pressure ranges, 200-600 MPa.
- Sintering can be done under different atmosphere like hydrogen, nitrogen or argon etc. and their impact on sintering density and densification parameter and sintering density can be further improved up 99% by hot compacting/hot isostatic pressure (HIP) method.
- Oxidation resistance and corrosion resistance tests under different atmosphere can be carried out.
- Above mentioned sintered pellets can be subjected to tensile, compression and impact, hardness at high temperature tests.
- White ash having silica, produced from the process can be used for aluminum matrix composite using stir-casting route, the produced MMC can be further characterized and mechanical behavior like tensile strength, compressive strength, impact strength tests can be done.

CHAPTER 7

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7. REFERENCES

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